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(54) Title: COMPOSITION OF CONTROLLED RELEASE OF PERFUMES AND FLAVOURS

(57) Abstract: The invention concerns an extruded composition capable of releasing a perfume or flavour when in contact with water. The invention composition comprises a water-soluble polymeric element and a perfuming or flavouring element, the latter containing a perfuming or flavouring ingredient, a hydrophilic, essentially spherical, hollow silica and optionally a surfactant. The composition can also comprise a dye as optional component. The invention composition is also useful as a means intended to protect a perfume or flavour from an aggressive medium. The invention also concerns the consumer articles containing, or associated with, said composition, in particular powder detergents or soap bars or tablets.



# COMPOSITION FOR CONTROLLED RELEASE OF PERFUMES AND FLAVOURS

#### Technical field

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The invention concerns an extruded composition capable of releasing a perfume or flavour when in contact with water.

The extruded composition according to the invention comprises:

- a) a water-soluble polymeric element, and
- b) a perfuming or flavouring element containing a perfuming or flavouring ingredient or
   composition, a hydrophilic hollow silica having a mean diameter comprised between
   150 μm and 350 μm, and optionally a surfactant.

The composition can also comprise a dye as optional component.

The invention also concerns the use of said composition as a means to protect a perfume or flavour from an aggressive agent or medium, such as a bleach in the case of a perfume. Moreover, the invention also concerns consumer articles or food containing, or associated with, said composition, in particular powder detergents or soap bars for the perfumery, and chewing gums or candies for flavours.

#### Prior art

Compositions which are able to release perfumes have become very common features nowadays in the perfume industry and are very frequently associated with detergents. However, said compositions present the inconvenient of needing large amounts of materials to deliver only a limited amount of perfume. One can cite the case of detergents wherein the amount of perfume, which is carried by and delivered from said compositions to the surface that is washed, is often small when compared with the total weight of the compositions.

In patent application EP 728804 there are disclosed compositions which tend to partially solve the problem mentioned hereinabove. The compositions described in said document are capable of containing up to 20% by weight of a perfume and also have the advantage of being of simple formulation, contrary to many other compositions disclosed in other prior art documents. More precisely, said compositions comprise a water-soluble

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polyvinyl polymer, i.e. a hydrophilic polymer, which contains up to 20% by weight of a fragrance. In order to incorporate into the hydrophilic polymer such an amount of perfume, which is in general a hydrophobic liquid, the latter is previously absorbed into a hydrophobic silica.

However, the compositions disclosed in EP 728804, despite their advantages, still do not represent an optimal solution to the mentioned problem as they deliver only up to a fifth of their weight of perfume in the best cases, i.e. 80% of their weight is lost in carrier materials which will be dispersed into the environment.

Therefore, there is still a need, especially for the detergent industry, for a composition which is capable of carrying and delivering high amounts of a perfuming ingredient in order to limit the total mass of the composition, and consequently the waste into the environment, while maintaining high performance of fragrance delivery.

A similar problematic is known in the flavour industry where there is a constant need for improving the loadings and delivery of flavour ingredients or compositions from the same type of compositions.

## Description of the invention

We have now surprisingly discovered that, by using a hydrophilic hollow silica to absorb a hydrophobic perfume or flavour, rather than a hydrophobic silica as in the prior art, it is possible to obtain a water-soluble composition of simple formulation and capable of carrying high amounts of perfume or flavour, thus limiting the amount of carrier or wastes which will be dispersed in the environment.

The extruded composition according to the invention comprises:

- a) a water-soluble polymeric element, and
- b) a perfuming or flavouring element containing:
  - i) a perfuming or flavouring ingredient or composition,
  - ii) a hydrophilic hollow silica having a mean diameter comprised between 150  $\mu m$  and 350  $\mu m$ , and
  - iii) optionally a surfactant.
  - The composition can also comprise a dye as optional component.

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The water-soluble polymeric element is not only intended to carry the other ingredients of the invention composition, but also to protect the integrity of the perfuming or flavouring element during the manufacture and the storage of the composition or consumer article according to the invention as described further below.

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An appropriate polymeric element for the invention must be able to be processed by extrusion, in order to allow the inclusion of the perfuming or flavouring ingredients into the polymer mass. Additionally, said polymeric element must be water-soluble, so that once the composition of the invention is in contact with a sufficient amount of water, said polymeric element will dissolve, thus liberating the perfuming or flavouring element and consequently releasing the perfume or flavour into the surrounding environment, for instance on a washed surface or yet in the mouth during the consumption of food. By "water-soluble polymeric element" it is meant here a polymeric element which is almost entirely, i.e. more than 90% of its total weight, dissolved in water at 90°C and at a 10% weight/weight concentration.

Ideally, the polymeric element consists of a water-soluble polymer.

As an example of a water-soluble polymer according to the invention, one can cite any grade of water-soluble polyvinyl alcohol or water-soluble partially hydrolysed polyvinyl acetate; said polymers having also the advantage of being biodegradable. Both polyvinyl alcohol and partially hydrolysed polyvinyl acetate are obtained by hydrolysis of polyvinyl acetate. Preferably, the water-soluble polymer of the invention has a percentage of hydrolysed acetate groups comprised between 80% and 99%, most preferably between 85% and 99%.

The polyvinyl alcohol and partially hydrolysed polyvinyl acetate are known to modify the viscosity of water when dissolved. Preferably the water-soluble polymer of the invention is such that, when dissolved at a concentration of 4% weight/weight in water at 20°C it provides solutions having a viscosity lower than 40 mPa's, more preferably lower than 20 mPa's.

Examples of commercially available water-soluble polymers, which are useful for the invention, are known under the tradename Mowiol<sup>®</sup> 10/98, Mowiol<sup>®</sup> 4/88, Mowiol<sup>®</sup> 8/88 (origin: Clariant, Germany) or yet Soltec<sup>®</sup> T10 (origin: Soltec Développement SA, France) or Solublon<sup>®</sup> (origin Aicello, Japan).

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More frequently, the water-soluble polymeric element will also contain a plasticiser in order to facilitate the extrusion process of the composition. One can cite, as non-limiting examples of plasticisers, water, glycerine, ethylene glycol, propylene glycol, dipropylene glycol or diethylene glycol. Preferably the plasticiser will be water or glycerine.

The amount of plasticiser present in the polymeric element may vary between 0% to 20%, preferably between 1% to 10%, the percentage being relative to the weight of the polymeric element.

The compositions of the invention may contain the polymeric element in an amount comprised between 40 % and 70 %, the percentages being relative to the total weight of the composition. In a preferred embodiment of the invention, the polymeric element is present in an amount comprised between 45 % and 55 %.

The perfuming or flavouring element contains two different ingredients, each of them having a precise function. The perfuming or flavouring element in the final extruded composition is essentially included or contained into the water-soluble polymeric element.

The first ingredient of the perfuming or flavouring element is a perfuming or flavouring ingredient or composition. It must be properly understood that a perfuming or flavouring composition, within the framework of the invention, designates a composition essentially comprising ingredients currently used in perfumery or flavour industry and able to impart an odour, or to improve, enhance or modify the odour or taste properties of the composition to which they are added. Usually, these ingredients will form a more or less complex mixture of volatile ingredients of natural or synthetic origin. The nature of these ingredients can be found in specialised books, e.g. in S. Arctander (Perfume and Flavor Chemicals, Montclair N.J., USA 1969), or in Fenaroli's Handbook of Flavour Ingredients, CRC Press or yet in Synthetic Food Adjuncts by M.B. Jacobs, van Nostrand Co., Inc. or similar textbooks of reference, and a more detailed description thereof is not warranted here. Additionally, useful perfuming or flavouring ingredients will be in a liquid form.

The amount of perfuming or flavouring ingredient present in the perfuming or flavouring composition may vary between 50% and 100%, preferably between 90% and

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100%, the percentage being relative to the weight of the perfuming or flavouring composition.

The perfuming or flavouring composition may further comprise one or more additional ingredients such as antioxidant agents, antibacterial or bacteriostatic agents, insect repellents or cosmetic or skin care ingredients.

As non-limiting examples of antioxidants one can mention Tocopherol, BHT (Butylhydroxytoluene) DLTDP (Dilaurylthiodipropionate). As cosmetic skin caring ingredients one can mention emollients, lanolin, esters of fatty acids with different mono, di and trivalent alcohols, vaseline oil, squalane, olive oil and others, moisturisers, amino acids, glycerine, sorbitol, polyvinylpyrrolidone, plant extracts such as aloe, hamamelis, horse chestnut, ginko biloba and many others and vitamins such as Vitamin C and its ester, Vitamin E acetate, Vitamin F (unsaturated fatty acids) and others. Regarding the antibacterials or bacteriostatics effect of the perfuming ingredient, it is useful to mention that the perfume itself may possess such effects.

The amount of additional ingredients present in the perfuming or flavouring composition may vary between 0% and 50%, preferably between 0% and 10%, the percentage being relative to the weight of the perfuming or flavouring composition.

According to the invention, the perfuming or flavouring ingredient or composition may be incorporated in the perfuming or flavouring element in an amount comprised between 20 % and 70 %, the percentages being relative to the total weight of the perfuming or flavouring element. Preferably, the perfuming or flavouring ingredient or composition is present in amounts comprised between 30 % and 70 %, even more preferably between 50 % and 70 %, the percentages being relative to the total weight of the perfuming or flavouring element.

The second ingredient of the perfuming or flavouring element is a particular type of silica, namely a hydrophilic hollow silica. The hollow silica is intended to contain or absorb the perfuming or flavouring ingredient or composition.

Useful hydrophilic hollow silica have an essentially spherical morphology and have a mean diameter comprised between 150  $\mu m$  and 350  $\mu m$ , preferably between 200  $\mu m$  and 300  $\mu m$ . The morphology of said hydrophilic hollow silica is thus similar to that of a micropearl.

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The size and the hydrophilicity of the hollow silica are important aspects of the latter. Indeed we have surprisingly found that by using such large hollow particles, instead of an amorphous silica or smaller silica particles, it was possible to extrude the composition of the invention without observing any appreciable leak of perfume or flavour from the silica, even when high amounts of perfume or flavour were incorporated within. The hydrophilicity of the hollow silica allows a better release into the surrounding environment of the perfume or flavour which is, in general, a hydrophobic liquid. The specific properties of the hollow silica used in the invention permit therefore to obtain an extruded composition capable of incorporating high amounts of perfume or flavour and also to release, or deliver into the surrounding environment, said perfume or flavour in an efficient manner.

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In addition to the above-mentioned properties, preferably the silica also has a high oil absorption capacity, i.e. it is capable of absorbing oil in more than two times its own weight. The oil absorption capacity may be measured using dioctylphthalate as reference compound, according to a method described in WO 99/49850, and is measured in g/100 g of silica or in ml/100 g of silica. Thus, the silica of the invention has, preferably, an oil absorption capacity comprised between 230 ml/100 g and 350 ml/100 g, or between 230 g/100 g and 350 g/100 g.

Said hydrophilic hollow silica is obtainable by spray drying of precipitated silica as described in WO 99/49850, the contents of this preparation there-described is incorporated herein by reference.

Examples of commercially available hydrophilic hollow silica useful to the invention are known under the tradenames Sipernat<sup>®</sup> 2200 (origin: Degussa, Germany) or Tixosil<sup>®</sup> 38X or 68 (origin: Rhodia, France).

The hydrophilic hollow silica may be incorporated in the perfuming or flavouring element according to the invention in an amount comprised between 30 % and 70 %, the percentages being relative to the total weight of the perfuming or flavouring element. Preferably, the hydrophilic hollow silica is present in amounts comprised between 30 % and 50 %, even more preferably between 30 % and 40 %, the percentages being relative to the total weight of the perfuming or flavouring element.

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The presence of a surfactant in the perfuming or flavouring element is optional, but preferred, and is intended to help the absorption of the hydrophobic perfume or flavour by the hydrophilic hollow silica. As non-limiting examples, the surfactant may be selected from the group consisting of polyalkyleneglycol ethers of a C<sub>1</sub>-C<sub>10</sub> alkanol, polyalkyleneglycol ethers of synthetic C<sub>9-20</sub> fatty alcohols and mono laurate esters of sorbitol condensed with polyethylene oxide. Preferably, the surfactant is selected from the group consisting of polysorbates, PPG/Buteth derivatives, and Pareth derivatives.

Examples of commercially available surfactants, which are useful to the invention, are known under the tradename Tergitol<sup>®</sup> 15-S-9 (C<sub>11-15</sub> Pareth-9) (origin: Union Carbide, USA), Tergitol<sup>®</sup> XD (PPG-24-Buteth-27) (origin: Union Carbide, USA) or Tween<sup>®</sup> 20 (origin: ICI, UK).

The surfactant may be incorporated into the perfuming or flavouring element according to the invention in an amount comprised between 0% and 10%, the percentages being relative to the total weight of the perfuming or flavouring element. Preferably, the surfactant is present in amounts comprised between 3% and 8%, the percentages being relative to the total weight of the perfuming or flavouring element.

The compositions of the invention may contain the perfuming or flavouring element in an amount comprised between 30% and 70%, the percentages being relative to the total weight of the composition. In a preferred embodiment of the invention, the perfuming or flavouring element is present in an amount comprised between 45% and 55% by weight, relative to the total weight of the composition.

It is thus possible to obtain compositions according to the invention having a very high load of perfume or flavour, i.e. up to approximately 40%. Such levels of perfume or flavour imply that, to deliver the same amount of perfume or flavour, a composition according to the invention needs half the weight of carrier when compared with a composition disclosed in EP 728804.

Compositions according to the invention having a perfume or flavour content comprised between 25% and 35% of the composition's total weight are preferred.

The compositions of the invention may further comprise, as optional component, a dye, preferably a hydrophilic one, such as a naphthalene or trityl derivative. Non-limiting examples of suitable dyes are Patent Blue V [trityl derivative: N-[4-[[4-

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dietheylamino)phenyl](5-hydroxy-2,4-disulfophenyl)methylene]2,5-cyclohexadien-1-ylidene]-N-ethylethanaminium, hydroxide, inner Salt or calcium Salt (2:1)], Food Green 3 [trityl derivative: benzenemethanaminium, N-ethyl-N-[4-[[4-[ethyl[(3-sulfophenyl)methyl]amino]phenyl](4-hydroxy-2-sulfophenyl)methylene]-2,5-

cyclohexadien-1-ylidene]-3-sulfo-, hydroxide, inner Salt or disodium Salt], Fuchsine [trityl derivative: 4-[(4-aminophenyl)(4-imino-2,5-cyclohexadien-1-ylidene)methyl]-2-methylbenzenamine monohydrochloride] or Orange 6 [naphthalene derivative: (disodium 7-hydroxy-8-(phenylazo)-1,3-naphthalenedisulfonate)].

The dye component may be incorporated into the composition of the invention in an amount comprised between 0% and 1%, the percentages being relative to the total weight of the composition. Preferably, the dye component is present in amounts comprised between 0.005% and 0.5%.

The composition of the invention may be obtained by extruding a mixture of its constituents. The extrusion may be performed according to a "master batch" method, i.e. addition into the extruder of a mixture of all the elements, or alternatively according to a "side feeding" method, wherein the polymeric element is introduced into the extruder and, downstream, combined with the perfuming or flavouring element.

As extruder devices there may be used single or double screw extruders. Examples of such extruders are described in Schwarz/Ebeling/Lüpke/Schelter: Kunststoffverarbeitung, 2. Ed., Vogel Verlag, 1983.

The extruder is equipped with a temperature regulation mechanism so that it is possible to maintain the temperature of the mixture in a range comprised between 90°C and 270°C, to form a molten mass. As the molten mass exits the extruder, it is cooled, using standard methods which do not need water, and it can be chopped into a multitude of granules by using an appropriate apparatus, to obtain a composition of the invention in a granular form. The size and diameter of the granules will depend on the opening of the exit nozzle and the stroke rate of the cutting apparatus; said size is preferably comprised between 0.1 mm and 10.0 mm; more preferably between 1 mm and 5.0 mm.

Different forms of the granules or flakes, optionally with an aesthetic function, e.g. flowers or stars etc, can be obtained by combining the adequate extruder opening with cutting devices.

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As anticipated above, the composition of the invention, preferably in the form of granules or flakes, may be contained in, or associated with, a consumer article such as in perfumery, a solid detergent in the form of tablets, a powder or a bar and in the case of flavours, a food.

Solid detergents, constitute another object of the invention and may be intended for textile or skin treatment, or also for cleaning dishes or varied surfaces, for industrial or household use.

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Tablets of detergents may be obtained according to any current method of production of such tablets.

Powder detergents according to the invention may be obtained by admixing the composition of the invention, in a granular form, with said powder. Thus, it will be obtained a powder detergent containing, as perfume dispenser, the composition of the invention.

Detergent bars, such as soaps, according to the invention may be obtained by extruding the composition of the invention, in a granular form, together with the detergent mass. Detergent bars represent an unexpected application of the invention compositions. Indeed the extrusion of soaps required very high pressures which generally break the most of the capsule or microsponge systems known. The invention's extruded granules or flakes resist to the pressure and shearing action of standard bar soap manufacturing extruders.

According to their compositions, detergents may be considered as aggressive media for perfumes. Indeed strong bases or bleaches, which may be present in the detergent formulation, can chemically degrade the perfume with the undesired consequence of changing the perfuming effect that the latter can confer to a treated surface. Said degradation may occur upon storage and/or upon use of the detergent. Generally, the degradation observed upon storage is more important due to the longer time of contact between the perfume and the detergent.

In the case of flavours, the compositions of the invention may be added to sweet applications such as chewing gums in particular. Chewing gum compositions are obtained by conventional methods, well known by a skilled person in the art.

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Now, we have surprisingly discovered that the invention compositions are also able to protect the perfume or flavour from chemical degradation. In the perfumery field, such degradation may be caused by some ingredients present in a detergent, such a protection being thus particularly effective during the storage of the invention's detergent. The protective action is believed to be due to the specific nature of the invention composition wherein the perfume is protected twice, by the polymer and by the hollow silica, from an accidental contact with aggressive ingredients of the medium, thus avoiding, minimizing or delaying the mechanisms responsible for the degradation. It is also possible that some interactions between the polymer or the silica and the perfume somehow stabilise the latter, making it less sensitive to the action of aggressive ingredients such as oxidants. Similarly in the case of flavouring ingredients, losses of volatile components from food products may produce undesirable variations in the taste and aroma of the products as perceived by the consumer. On the other hand, losses of volatile components might occur through the conversion of certain flavour materials into unwanted less desirable or tasteless chemicals by their interaction with reagents present in the environment. Oxygen is an example of this type of reagent as it promotes the conversion of several labile flavour materials of current and critical utilisation in the industry. Therefore, it is always useful to be able to efficiently protect flavouring ingredients from degradation, during storage and before their consumption.

Consequently, another object of the invention is the use of a composition according to the invention as a means to protect a perfume or flavour from an aggressive medium or agent, such as a bleach, oxygen or a strong base.

Another advantage of the invention resides in the fact that the consumer products containing a composition according to the invention are able to impart a more intense fragrance or flavour, unlike the effect obtained with a consumer product perfumed or flavoured strictly with the fragrances or flavours as such.

Moreover, we have also discovered that in the case of perfumes, the abovementioned protective action is also effective upon use of the detergent. Indeed, by carefully choosing the size of the granule, or the type of water-soluble polymer, it is possible to adjust the time needed by a composition according to the invention to release the perfuming ingredient or composition, once said composition is in contact with water,

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such as during the use of the detergent. We have thus found that it is possible to obtain compositions according to the invention that may deliver the perfume to the treated surface in a period of time comprised between 0.5 and 30 minutes, or even 60 or 90 minutes after the contact with water.

This behaviour is very attractive since it allows to use a composition according to the invention either for a fast release or for a slow release of the perfume.

The slow release of the perfume is particularly attractive as it allows the delivery of the perfume on the treated surface once the most aggressive ingredients of the detergent have been washed out or consumed, thus minimising the chance of the perfume degradation upon use of the detergent.

Therefore, due to the particular behaviour of the invention composition which allows to minimise the chance of the perfume degradation upon storage and use, to choose the time of release of the perfume, and eventually also the kinetic of said release, yet another object of the invention is a process for the perfuming of a surface or a process for intensifying, prolonging or deferring the diffusion effect of the characteristic fragrance of a perfume on a surface, characterised in that said surface is treated in the presence of a composition according to the invention.

In the case of flavours, the compositions of the invention also proved to be very advantageous as they allow to provide a particularly intense flavouring effect compared with the case wherein the flavour ingredient or composition is used in a free form.

The invention will now be described in further detail by way of the following examples wherein temperatures are given in degrees Celsius, and the abbreviations have the usual meaning in the art.

25 <u>Example 1</u>

# Manufacture of extruded compositions according to the invention

#### The perfuming element

A perfuming element was obtained by admixing, in the given sequence, in a beaker the following ingredients to allow a total absorption of the fragrance into hollow silica:

Fragrance*	325 g
Tween® 20 1)	25 g
Sipernat <sup>®</sup> 2200 <sup>2)</sup>	<u>150 g</u>
	500 g

- 1) polyoxyethylene(20)sorbitan monolaurate; origin: ICI, UK
- 2) hollow silica, origin: Degussa, Germany
- \* Fragrance composition

10	Ingredient	Part by weight %
	4-Dimethyl-3-cyclohexene-1-carbaldehyde	10
	Benzyl Salicylate	10
	Lilial <sup>®</sup> (origin : Givaudan)	10
	3-(3-Isopropyl-1-phenyl)butanal	10
15	(+-)-3-(4-isopropylphenyl)-2-methylpropanal	10
	2-Methyl undecanal	10
	Eugenol	10
	Benzyl acetate	10
	Hexyl salicylate	10
20	Phenylethyl acetate	<u>10</u>
		100

## The extruded compositions

- a) 250 g of above perfuming element were mixed with 250 g of a polyvinyl alcohol (Soltec® T10, Soltec SA, France) and with 1 g of a dye (Vibracolor blue PBL 15/3 L, Ciba AG, Switzerland) to obtain blue coloured granules.
  - Finally the whole mixture was extruded in a 2-screw Brabender (Germany) laboratory extruder with the following temperatures :
- Extruder zone 1 = 200°; zone 2 = 220°, zone 3 = 180°. With an extruder outlet of 5 mm, a string of the same diameter was extruded and immediately cut to obtain granules of the same diameter containing 32.5% of fragrance.

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b) 200 g of the above perfuming element were mixed with 400 g of polyvinyl alcohol (Soltec<sup>®</sup> T10, Soltec SA, France) and with 0.2 g of a dye (Vibracolor yellow PYE 1-L, Ciba AG Switzerland) and 0.8 g of another dye (Vibracolor green PGR 7-L, Ciba AG Switzerland) to obtain a green colour.

The extrusion was performed with the same conditions as part a) above but with an extruder outlet allowing the formation of a flat film with 0.3 - 0.4 mm thickness containing 20% fragrance. The film was immediately cut in small flakes of 4 - 5 mm size.

#### Example 2

Stability of PVOH extruded fragrance in a detergent powder

Various invention compositions, obtained according to example 1b), were admixed with a standard European detergent powder with percarbonate-TAED bleaching system. After two weeks storage at 37°C and 70% of relative humidity, the invention compositions were separated from the detergent, dissolved in water and diluted with acetonitrile and 2-heptanone. 1 g of the solution thus obtained was dried over 5 g of Na<sub>2</sub>SO<sub>4</sub> and the residual liquid phase was analysed by a standard GC-MS analytical method to determine the residual quantity of the various fragrant aldehydes. The results were compared with those obtained with a reference detergent, stored for two weeks at 3°, on which the perfume was directly sprayed-on. The results are summarised in Table 1.

Table 1: Percentage of the total aldehyde recovered after storage

Aldehyde	Reference (sprayed-on)	Granules with 20% perfume loading	Granules with 25% perfume loading	Granules with 32.5% perfume loading
2-Methyl-undecanal	0%	28%	31%	41%
3-(3-Isopropyl-1-phenyl)butanal	6%	44%	44%	50%
Cyclosal	5%	28%	28%	31%
Lilial®	6%	35%	34%	37%
Time needed for full perfume release	<10 minutes	~30 minutes	~30 minutes	>30 minutes

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As it can be noticed, when compared to the reference, the granules according to the invention are always able to deliver higher quantities of fragrant aldehydes by decreasing considerably the degradation of the latter, and are also able to delay the release of said aldehydes in water, thus decreasing also the probability of contact between said aldehydes and the bleaches.

#### Example 3

## Manufacture of extruded compositions according to the invention

A perfuming element was obtained by admixing, in the given sequence, in a beaker, the following ingredients to allow a total absorption of the fragrance into hollow silica:

Ingredients grams
Fragrance 1) 360
Sipernat® 2200 2) 200

1) fleur de menthe 68528 E : origin : Firmenich SA, Geneva, Switzerland

2) hollow silica; origin: Degussa, Germany

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The perfuming element was mixed with 337 g of polyvinyl alcohol (V03/180, origin: Erkol SA, Spain), 60 g of water and with 3 g of dye (1 g of vibracolor vert PGR7L et 2 g of vibracolor jaune PYE13L; origin: Ciba AG, Switzerland) to obtain green coloured granules.

Finally, the whole mixture was extruded in a 2-screw PRISM-EuroLab extruder with the following process parameters: temperature 108°; pressure 10-15x10<sup>5</sup> Pa; screw rpm 180; die hole 1.5 mm.

The capsules obtained were characterised by 26 to 28% by weight of perfume load relative to the total weight of the capsules.

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#### Example 4

Manufacture of extruded compositions according to the invention

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A perfuming element was obtained by admixing, in the given sequence, in a beaker, the following ingredients to allow a total absorption of the fragrance into hollow silica:

	<u>Ingredients</u>	grams
5	Fragrance 1)	187
	Sipernat <sup>®</sup> 2200 <sup>2)</sup>	93

## 1) fragrance composition:

	<u>Ingredients</u>	Parts by weight
10	Verdox® (origin : IFF, USA)	42.6
	Allyl heptanoate	25.5
	Hexyl salycilate	14.9
	Phenoxy isobutyrate	17.0
	Total	100.0

2) hollow silica: origin: Degussa, Germany

The perfuming element was mixed with 654 g of polyvinyl alcohol (V03/180, origin: Erkol SA, Spain) and 66 g of water.

Finally, the whole mixture was extruded in a 2-screw PRISM-EuroLab extruder with the following process parameters: temperature 108°; pressure 10-15x10<sup>5</sup> Pa; screw rpm 180; die hole 1.5 mm.

The obtained granules were loaded with 15% of fragrance.

25 <u>Example 5</u>

#### A soap bar containing a composition according to the invention

The granules and flakes from example 3 were incorporated at 1% in a classical translucent soap base type 1984 from Uniqema (The Netherlands).

The soap extrusion was performed at a temperature comprised between 45° and 50°C on a Beck laboratory extruder type BV45 made by Ehrismann SA, Switzerland. Soap bars of

90 g were pressed on a RMT laboratory soap press (RMT Ltd. Great Britain). The granules and flakes were not broken and gave a pleasant visual effect to the soap bar

#### Example 6

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# Manufacture of extruded compositions according to the invention

A flavouring element was obtained by admixing, in the given sequence, in a beaker, the following ingredients to allow a total absorption of the flavour into hollow silica:

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<u>Ingredients</u>	grams
Flavour 1)	10
Cooling agent 2)	40
Sipernat <sup>®</sup> 22S <sup>3)</sup>	93

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- 1) 758810 01T; origin: Firmenich SA, Geneva, Switzerland
- 2) 927875; origin: Firmenich SA, Geneva, Switzerland
- 3) hollow silica; origin: Degussa, Germany
- The flavouring element was admixed with 870 g of polyvinyl alcohol (V03/180, origin: 20 Erkol SA, Spain) 60 g of water, 5 g of Citrem® (origin: Danisco, Denmark) and 5 g of fractionated coconut oil (origin: Stearineric Dubois).

Finally, the whole mixture was extruded in a 2-screw PRISM-EuroLab extruder with the following process parameters : temperature  $130^{\circ}$ ; pressure  $10\text{-}15\text{x}10^{5}\,\text{Pa}$ ; screw rpm

180; die hole 1.5 mm. 25

#### Example 7

# Application of flavoured capsules according to the invention in chewing gums

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A chewing gum was formulated with the following ingredients:

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	<u>Ingredients</u>	Parts by weight
	Gum base 1)	250
	Sorbitol powder	523
	Sorbitol syrup	150
5	Glycerine	65
	Aspartame®	2
	Acesulfam-K	2
	Flavoured capsules 2)	8
	Total	1000

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- 1) L.A. Dreyfus Nova
- 2) prepared according to Example 6

The gum base was added to a classical gum mixer and mixed until homogeneous. Half of the sorbitol powder and half of the syrup were added and the whole mixture was mixed for 5 to 8 minutes. The glycerine was added and mixed one minute. The flavoured capsules were added and mixed for 4 to 5 minutes. The final mixture was removed from mixer, shape cut and wrap.

The chewing gum obtained proved to have an increased flavour impact without bitterness and longer lasting flavour duration compared with similar products with free flavours.

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## **Claims**

- 1. A composition comprising:
- a) a water-soluble polymeric element, and
- b) a perfuming or flavouring element containing:
  - i) a perfuming or flavouring ingredient or composition,
  - ii) a hydrophilic hollow silica having a mean diameter comprised between 150  $\mu m$  and 350  $\mu m$ , and
  - iii) optionally a surfactant.

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- 2. A composition according to claim 1, wherein the water-soluble polymeric element consists of a water-soluble polymer.
- 3. A composition according to claim 2, wherein the water-soluble polymer is
  a water-soluble polyvinyl alcohol or a water-soluble partially hydrolysed polyvinyl acetate.
  - 4. A composition according to claim 2, wherein the polymeric element further comprises a plasticiser selected from the group consisting of water, glycerine, ethylene glycol, propylene glycol, dipropylene glycol or diethylene glycol.
  - 5. A composition according to claim 1, comprising a perfuming element containing a perfuming composition.
  - 6. A composition according to claim 5, wherein the perfuming composition comprises at least one perfuming ingredient and one or more additional ingredients selected from the group consisting of antioxidant agents, antibacterial or bacteriostatic agents, insect repellents or cosmetic or skin care ingredients.
- 7. A composition according to claim 1, wherein the hydrophilic hollow silica has an oil absorption capacity comprised between 230 ml/100 g and 350 ml/100 g.

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**8.** A composition according to claim 1, wherein the surfactant is selected from the group consisting of polyalkyleneglycol ethers of a C<sub>1</sub>-C<sub>10</sub> alkanol, polyalkyleneglycol ethers of synthetic C<sub>9-20</sub> fatty alcohols and mono laurate esters of sorbitol condensed with polyethylene oxide.

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- 9. A composition according to claim 1, wherein the perfume content is comprised between 25% and 35% of the composition's total weight.
  - 10. A composition according to claim 1, further comprising a dye.

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- 11. A composition according to any one of claims 1 to 10, in a granular form wherein the granules have a size preferably comprised between 0.1 mm and 10,0 mm.
- 12. Use of a composition according to any one of claims 1 to 11, as a means
  intended to protect a perfume or flavour from an aggressive medium
  - 13. A process for the perfuming of a surface or a process for intensifying, prolonging or deferring the diffusion effect of the characteristic fragrance of a perfume on a surface, characterised in that said surface is treated in the presence of a composition according to anyone of claims 1 to 11.
  - 2 ,
  - 14. A consumer article or a food containing a composition according to any one of claims 1 to 11.
- 25 15. A consumer article according to claim 14, in the form of tablets, a powder or bar detergent.
  - 16. A food according to claim 14, in the form of a chewing gum.

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European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,

PCT/IB 03/02630 A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A61L9/04 A61L9/05 A23L1/22 A23L1/00 According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) A61L A23L Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data, PAJ C. DOCUMENTS CONSIDERED TO BE RELEVANT Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. Category ° EP 1 048 687 A (INT FLAVORS & FRAGRANCES 1-7,10,Y 12-15 INC) 2 November 2000 (2000-11-02) paragraphs '0012!-'0015!, '0024!-'0027!, '0029!-'0038! WO 99 49850 A (RHONE POULENC CHIMIE ; VIOT 1-7,10,Υ 12 - 15JEAN FRANCOIS (FR)) 7 October 1999 (1999-10-07) cited in the application page 2, line 20-36 page 3, line 18-21 page 6, line 23-28 -/--Further documents are listed in the continuation of box C. Patent family members are listed in annex. X Special categories of cited documents : later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the "A" document defining the general state of the art which is not considered to be of particular relevance invention \*E\* earlier document but published on or after the international "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to filing date \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the off. "O" document referring to an oral disclosure, use, exhibition or in the art. "P" document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of mailing of the international search report Date of the actual completion of the international search 14/10/2003 7 October 2003

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